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## PATENT ABSTRACTS OF JAPAN of D6

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(71)Applicant : SHIN ETSU CHEM CO LTD

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ISHIHARA TOSHINOBU  
YAMAMOTO AKIRA

## (54) PRODUCTION OF TERTIARY HYDROCARBONSILYL COMPOUND

## (57)Abstract:

PURPOSE: To safely and readily obtain the titled compound useful as a special silylating agent used for synthesizing medicines without using dangerous Li compounds, by reacting a Grignard reagent with a specific organosilicon compound in an organic solvent.

CONSTITUTION: (A) A Grignard reagent expressed by the formula  $R_1MgX$  ( $R_1$  is tertiary hydrocarbon;  $X$  is halogen), e.g. tert-butylmagnesium chloride, is reacted with (B) an organosilicon compound expressed by the formula  $AmR_2nSiH_{4-m-n}$  [ $R_2$  is (substituted) monofunctional hydrocarbon;  $a$  is alkoxy;  $m$  is 1,2 or 3;  $n$  is 0, 1 or 2;  $m+n \leq 3$ ], e.g. dimethylmethoxysilane, in an organic solvent, preferably THF, preferably at  $40W100^\circ C$  in an inert atmosphere to afford the aimed compound, e.g. tert-butylethoxysilane.

## LEGAL STATUS

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## Patent Abstracts of Japan

PUBLICATION NUMBER : 62022790  
PUBLICATION DATE : 30-01-87

APPLICATION DATE : 24-07-85  
APPLICATION NUMBER : 60163548

APPLICANT : SHIN ETSU CHEM CO LTD;

INVENTOR : YAMAMOTO AKIRA;

INT.CL. : C07F 7/18 C07F 7/08

TITLE : PRODUCTION OF TERTIARY HYDROCARBONSILYL COMPOUND

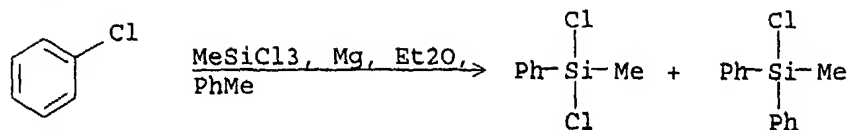
ABSTRACT : PURPOSE: To safely and readily obtain the titled compound useful as a special silylating agent used for synthesizing medicines without using dangerous Li compounds, by reacting a Grignard reagent with a specific organosilicon compound in an organic solvent.

CONSTITUTION: (A) A Grignard reagent expressed by the formula  $R^1MgX$  ( $R^1$  is tertiary hydrocarbon; X is halogen), e.g. tert-butylmagnesium chloride, is reacted with (B) an organosilicon compound expressed by the formula  $A_mR^2_nSiH_{4-m-n}$  [ $R^2$  is (substituted) monofunctional hydrocarbon; a is alkoxy; m is 1, 2 or 3; n is 0, 1 or 2;  $m+n \leq 3$ ], e.g. dimethylmethoxysilane, in an organic solvent, preferably THF, preferably at 40–100°C in an inert atmosphere to afford the aimed compound, e.g. tert-butylethoxysilane.

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feed of MeSiCl<sub>3</sub> to give Ph<sub>2</sub>SiMeCl/PhMeSiCl<sub>2</sub> in controllable ratios.

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NOTE: continuous process, ratio of products depends on ratio of reactants

REFERENCE COUNT: 9 THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

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ACCESSION NUMBER: 138:4688 CASREACT

TITLE: Method of preparing triphenylsilanol by phenylation of chloro(phenyl)silanes with phenylmagnesium chloride and subsequent reaction with water in mixed THF-toluene solvent

INVENTOR(S): Zhun, V. I.; Zhun, A. B.; Polivanov, A. N.; Chernyshev, E. A.

PATENT ASSIGNEE(S): Obshchestvo s Ogranichennoi Otvetstvennost'yu NPP "MAGNOS", Russia

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DOCUMENT TYPE: Patent

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RU 2174124	C2	20010927	RU 1999-125791	19991203
PRIORITY APPLN. INFO.:			RU 1999-125791	19991203

AB Triphenylsilanol is prepared by reaction of a phenylchlorosilane, e.g., Ph<sub>2</sub>SiCl<sub>2</sub> or PhSiCl<sub>3</sub>, with H<sub>2</sub>O in an organic solvent such that the phenylchlorosilane is treated with PhMgCl in a mixture of THF and toluene, and then the reaction mixture is treated with H<sub>2</sub>O in the same solvents, whereupon the desired product is isolated; the THF to toluene ratio used ranges from 1:3 to 3:1 by volume, resp. In an example, treating 500 g Ph<sub>2</sub>SiCl<sub>2</sub> with PhMgCl generated in situ from 225 g PhCl and Mg in a mixture of 250 mL THF and 250 mL PhMe and holding the mixture at room temperature 6 h followed by treatment with H<sub>2</sub>O gave 94.2% Ph<sub>3</sub>SiOH. This method makes it possible to prepare the desired product without isolation of Ph<sub>3</sub>SiCl from the reaction mixture followed by treatment with H<sub>2</sub>O and recovery of final product and also to use a mixture of solvents, thus causing greater process selectivity.